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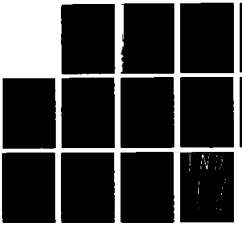

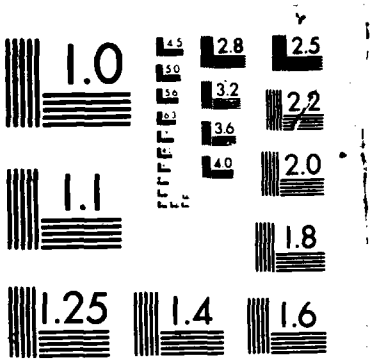

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MICROMECHANISMS OF FATIGUE CRACK GROWTH AND FRACTURE TOUGHNESS IN METAL MATRIX COMPOSITES

Technical Report for the Period 4/29/86 to 7/31/87

Prepared For

Office of Naval Research
800 North Quincy St.
Arlington, VA 22217

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Fatigue crack growth and fracture toughness of seven combinations of aluminum matrix alloys reinforced with particulate silicon carbide have been measured. The matrix alloys used were of both the work hardening and heat treatable varieties. Two of the alloys were made by mechanical alloying--powder metallurgy fabrication methods and the remainder were made by ingot metallurgy. Composites were tested in both as-received (extruded) and peak-aged conditions. Fractograpny was done for all the composites, as were cross sections to examine surface roughness.

The material with the largest fracture toughness value was 2014+15v/o SiC -- peak aged with 21 MPa$\sqrt{m}$, while that with the smallest was IN-9052+15v/o SiC with 9 MPa$\sqrt{m}$. Fracture toughness of the remainder of the alloys ranged between 12 and 18 MPa$\sqrt{m}$. Comparisons of the effects of matrix alloying and heat treatment and volume fraction of SiC have been made.

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THE FRACTURE OF ALUMINUM ALLOYS REINFORCED WITH SILICON CARBIDE

D. L. DAVIDSON

BACKGROUND

Aluminum alloys have proven to be excellent materials with which to construct light weight, strong structures. One of the major limitations of these alloys is their low elastic modulus, E = 70 GPa. By introducing silicon carbide with a modulus of E = 470 GPa, the composite modulus may be increased to E = 80 -120 GPa. This useful increase in modulus has resulted, however, in a loss of fracture toughness, which is highly undesirable.

Program Objectives

The objectives of our work on these composites is to determine the fracture characteristics of aluminum alloys reinforced with particulate silicon carbide, and seek answers to the following questions:

1. What is causing the fracture toughness of the composites to be lower than the unreinforced alloys?

2. What can be done to increase fracture toughness of the composite?

3. Can a combination of alloy composition, SiC volume fraction, manufacturing technique, etc., be found which optimizes the fracture toughness?

4. How does the mechanism of fatigue crack growth through the composites compare with unreinforced alloys?

Investigative Approach

The approach we have formulated mainly addresses question 3. However, it should be possible to answer the other questions posed above during the course of the investigation. Particulate size and volume fraction, matrix composition and ageing treatment, and manufacturing method were determined as being the important variables.

EXPERIMENTAL AND ANALYSIS PROCEDURES

Fracture Screening Tests

The fatigue and overload fracture characteristics of the composite combinations chosen were determined by testing samples of experimental materials obtained commercially. A computer controlled fatigue machine, automated by Fracture Technology Associates, was used to start a crack from the machined notch in compact tension specimens of the design shown in Fig. 1. Crack length was determined by the compliance method, using a calibrated crack mouth opening gauge. After crack initiation at $\Delta K = 8$ MPa$\sqrt{m}$, the cyclic load was reduced in steps of about 0.05 MPa$\sqrt{m}$ until a crack growth rate of approximately 1×10^{-9} m/cy was reached, approximating the threshold condition for fatigue crack growth. Subsequently, the load was held constant, and as the crack grew, ΔK increased until the point of rapid crack growth was reached, and the specimen fractured. Minimum load/maximum load (R ratio) was held constant at 0.1. The load and crack length at point of final failure allowed computation of the fracture toughness, K_{IC}. Duplicate tests were performed on all but one of the composites examined.

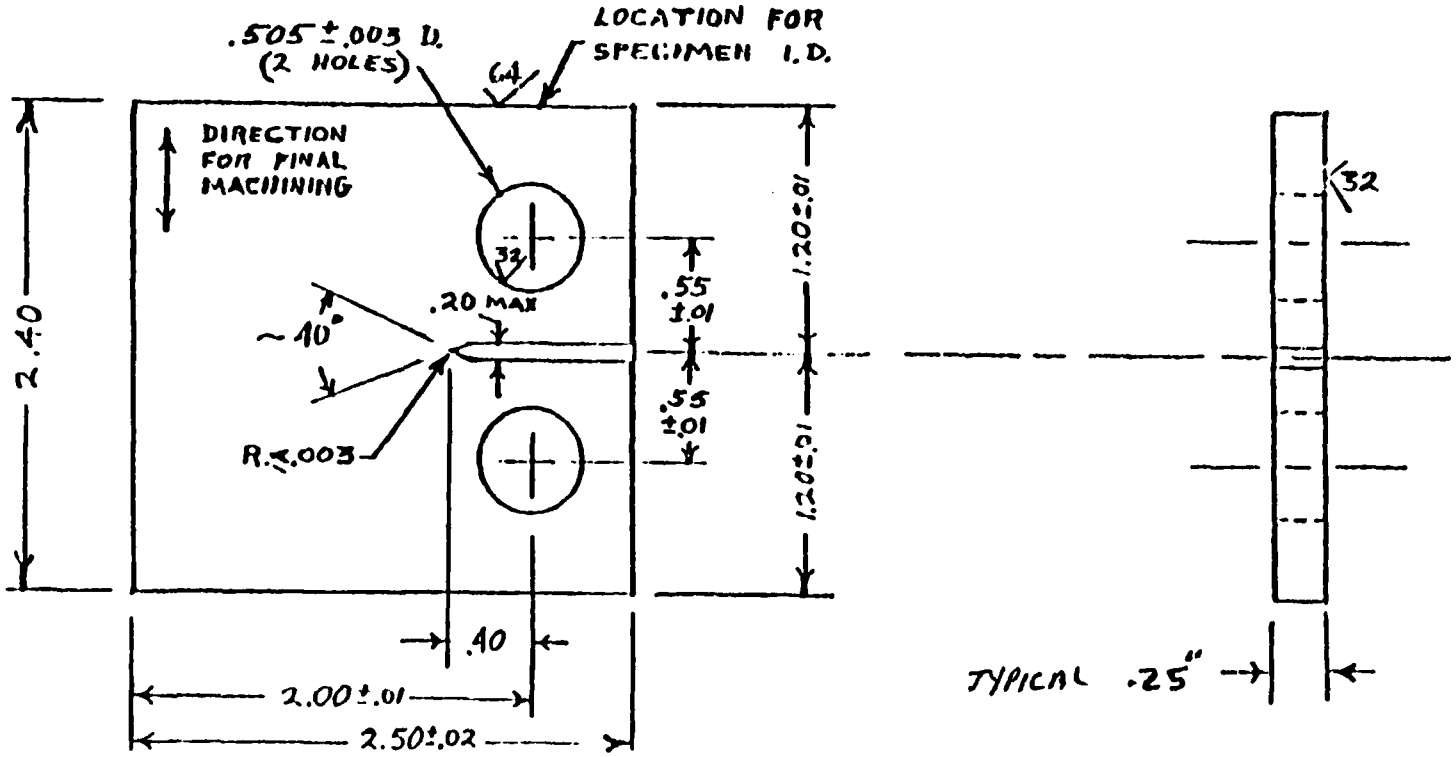

Figure 1. Specimen Design Used in Automated Fatigue Machine for Crack Growth Rate and Fracture Toughness Determinations

Crack Tip Characterization -

On selected composites, fatigue cracks were initiated, using a manually controlled laboratory machine, in single edge notch specimens which were designed also to fit a special cyclic loading stage for the scanning electron microscope. The gauge section of these specimens, 20 x 3 mm, was polished and etched lightly to reveal clearly the SiC particles. Periodically, cracked specimens were transferred from the laboratory machine to the scanning electron microscope cyclic loading stage for high resolution examination. Occasionally, some cycling was done in the SEM to observe the processes of crack growth, particularly as it related to interactions between SiC particles and the fatigue crack. Photographs of the crack tip region were obtained at maximum and minimum cyclic load as ΔK was increased from near threshold values to the onset of rapid fracture. Since the major part of the specimen loading was done in the laboratory fatigue machine, the results obtained are considered to have been obtained in laboratory air having about 50% humidity (12,000 ppm water vapor).

The stereoimaging technique was used to measure displacements within a 50 to 100 μm region centered on the crack tip. From gradients of these displacements, axial and shear strains were computed. Crack opening displacements in both Mode I and Mode II were obtained directly from the displacements. Stereoimaging was used also to determine the crack opening load directly at the crack tip.

Material Characterization -

Stress-strain curves were measured on selected compositions of the composites, using thin film, bonded strain gages to measure strain. Particle size distributions were determined from optical, scanning, and transmission

electron micrographs using a Dapple Image Analysis System. A very wide range of magnifications was required in order to completely characterize the particulates found in these materials. Particles sizes covering up to four decades in diameter were not exceptional. Homogeneity of particle distribution was also determined from metallography of these samples. Transmission electron micrographs that were used revealed the dislocation substructure of the matrix alloys and the presence of smaller particles.

Fractography -

Extensive fractographic examination was performed using mainly the scanning electron microscope, but occasionally, two-stage plastic, carbon replicas were used, together with transmission electron microscopy, for higher resolution. Micrographs were made from cross-sections of fracture surfaces in order to measure the roughness of the fracture patns and the degree of void formation and SiC fracture beneath the main fracture path.

MATERIALS:

Composites are manufactured by a variety of methods, including:

IM - ingot metallurgical practice,

MA - mechanical alloying together with powder consolidation methods,

PM -more traditional mixing techniques combined with powder metallurgical consolidation methods.

Each method of producing these composites has advantages and disadvantages. We have investigated aluminum alloys reinforced with particulate SiC made by both IM and MA methods.

Silicon Carbide -

Although it is possible to control the volume fraction of SiC in the finished composite, the size of the particulate cannot be specified to commercial fabricators because of: [1] limitations in obtaining SiC of the size desirable, [2] the inability to control particle size during manufacture (particularly in the MA process) and [3] the costs associated with controlling particle size. Therefore, careful control of the size of SiC in commercial composites is impractical.

In practice, it is necessary to obtain composites from fabricators and then characterize the SiC particle content. In fact, SiC particles in commercially available materials have a size distribution. Measurements of the SiC size distribution have proved to be difficult and expensive, and from a mechanistic view point, understanding the effect of a distribution of particle sizes is considerably more difficult than for singular sized particles. Our approach has been to accept these problems rather than attempt to make model materials, because when these composites are used in real structures, fabrication methods used in their manufacture will preclude monosized SiC particles.

Materials made by the IM process resulted in SiC particles with a larger mean size, but with a smaller size distribution, than composites made by the MA process. The MA process is the only one by which a relatively large volume fraction of very small SiC particles could be obtained. Thus, the particle sizes commercially obtainable were inseparably linked to the composite fabrication process.

Silicon carbide at room temperature exists in both the alpha and beta phases. The alpha phase has numerous polymorphs (crystallographic variations of the same composition). Neither the phase nor the polymorph of SiC is usually specified for composites because any change in properties which these variations might cause are considered to be minor compared to all the other factors. SiC extracted from the composites had a very dark appearance, indicating the presence of impurities or, perhaps, an excess of carbon.

Alloys - Aluminum is alloyed in compositions which can be strengthened either through work hardening or precipitation hardening. Mechanical characteristics of the matrix alloy are likely to have a large influence on composite properties, since the matrix is 75 to 85 % of the volume fraction of the composite. Matrix alloys strengthened by both precipitation and work hardening have been examined as variables. Table 1 lists the materials investigated as a part of this program, together with the descriptor used, hardening characteristic and volume fraction of particulate SiC in each material.

Comparisons - The materials investigated allow determination of the effect of manufacturing method, alloy composition, hardening characteristics, SiC volume fraction, and (to a lesser degree) particle size, on fatigue crack growth and fracture toughness of these composites. Changes caused by each of these variables can be determined, somewhat independently, by comparison of the various materials examined.

TABLE 1

MATERIALS EXAMINED

Process Type	Matrix Alloy	Volume Fraction SiC (%)	Age Hardening Alloy?
MA	IN-9052	15	No
IM	Al-4Mg	15	No
MA	IN-9021	14	Yes
IM	2014	15	Yes
IM	2014	25	Yes
IM	2024	15	Yes
IM	7475	15	Yes

MA = Mechanically alloyed: Manufactured by Novamet, Huntington, WV

IM = Ingot metallurgy: Manufactured by Dural, San Diego, CA

EXPERIMENTAL RESULTS:

Screening tests on a large number of materials have been completed, but detailed analysis of each material is still in progress. This section will present most of the results thusfar obtained.

Fatigue and Fracture Toughness Testing - Representative fatigue crack growth curves are shown in Fig. 2, which compares two materials having a large difference in fracture toughness. As may be seen, the slopes of the two curves are somewhat different, as is the K value at which rapid crack growth begins. The threshold stress intensity factor for fatigue crack growth has been estimated for each of these materials, even though a strictly valid threshold test may not have been conducted. For some materials, the crack growth curves are very straight, giving a long Stage II, or "Paris region," over almost the entire range in ΔK between threshold and final fracture. Several of the materials exhibited a sigmoidal shaped growth rate curve, more typical of unreinforced aluminum alloys. Values of the derived constants in the equation for Stage II crack growth rate

$$da/dN = B\Delta K^S \qquad (1)$$

are given in Table 2, together with estimated ΔK threshold values.

Fracture Toughness - Values of the fracture toughness, K_{Ic}, were obtained from the last load and crack length measurements obtained during the fatigue crack growth test. Since shear lips did not develop on the specimens while under cyclic loading, or during this final fracture event, constraint at the crack tip has been assumed to be of a nearly "plane strain" nature, resulting in a valid measurement of fracture toughness. Values of fracture toughness determined in this manner are shown in the last column of Table 2;

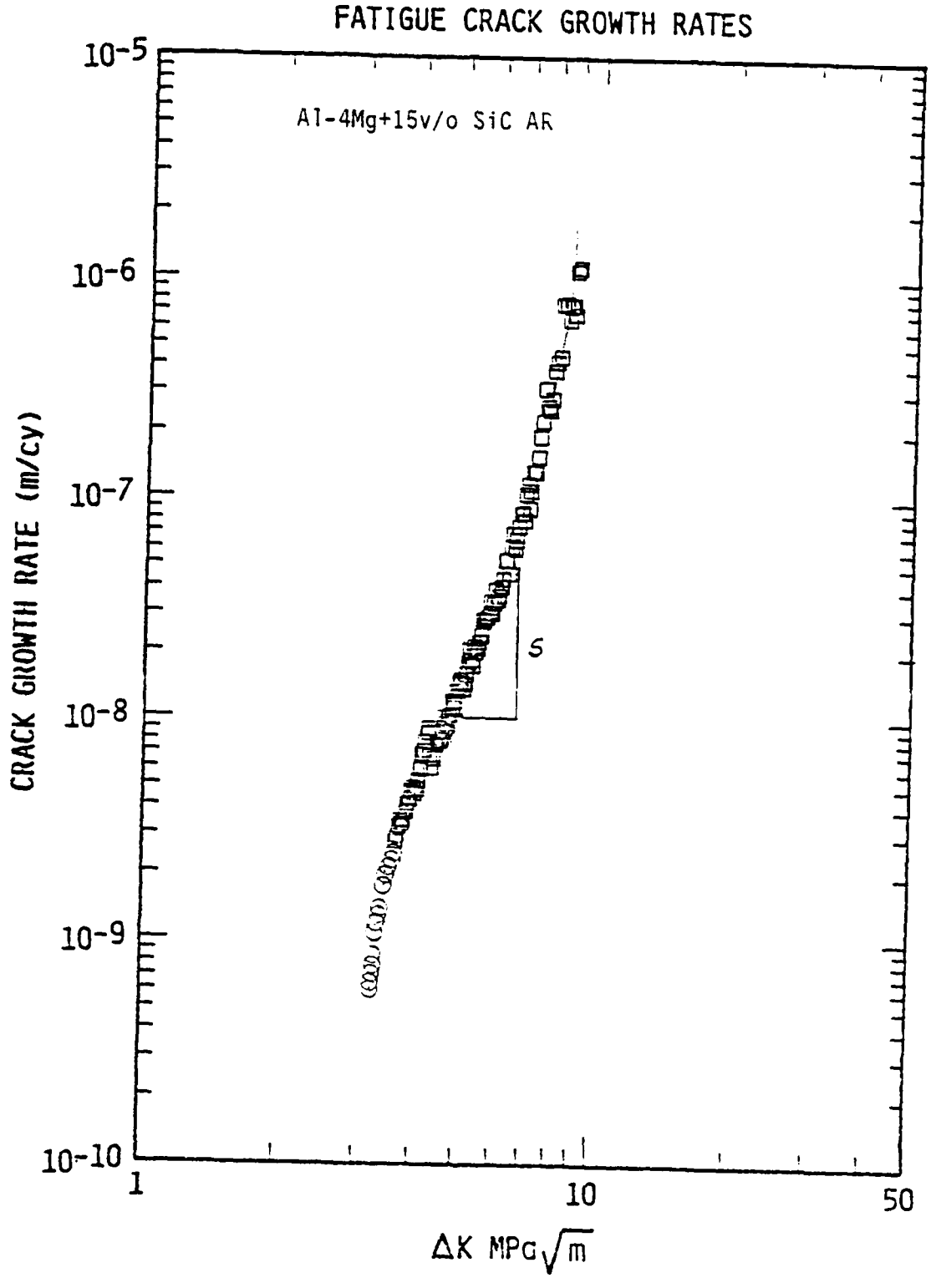

Figure 2. Representative Fatigue Crack Growth Curves Showing Definitions of the Terms Used to Describe It

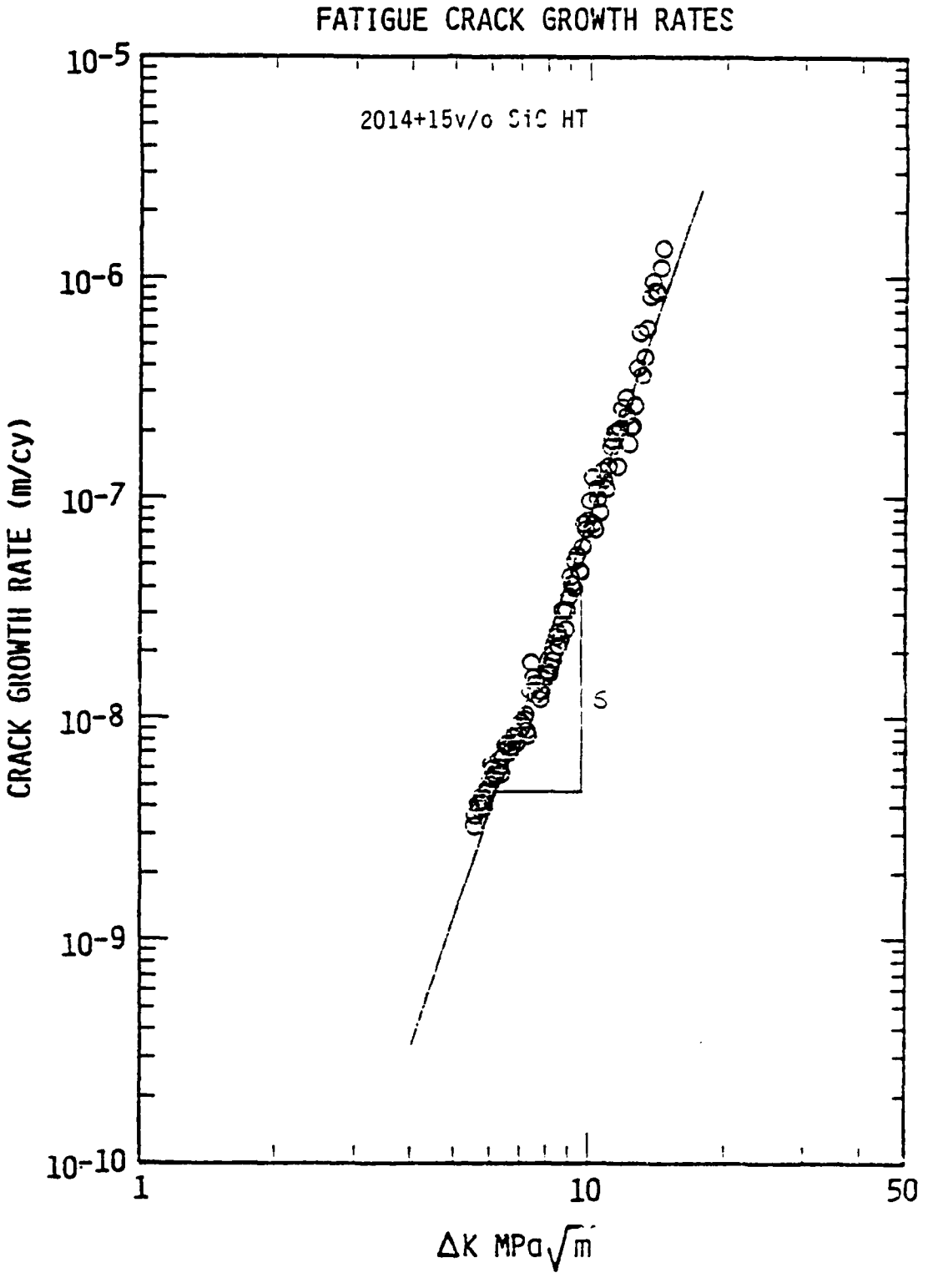

Figure 2 (Cont'd). Representative Fatigue Crack Growth Curves Showing Definitions of the Terms Used to Describe It

TABLE 2

FATIGUE AND FRACTURE TOUGHNESS RESULTS

Alloy No.	Designation	Condition	B m/cy	s	ΔK_{th} MPa$\sqrt{m}$ *	K_{Ic} MPa$\sqrt{m}$
1	IN-9052+15v/o	AR	1.1×10^{-12}	6.0	4.1	8.7, 9.1
2	AL-4Mg+15v/o	AR	1.4×10^{-13}	6.2	4.0	12.2, 12.2
3	IN-9021+14v/o	AR	1.1×10^{-12}	5.2	3.3	13.4
4	IN-9021+14v/o	PA	1.6×10^{-11}	4.4	3.0	13.8
5	2014+15v/o	AR	2.1×10^{-12}	4.6	3.6	13.8, 14.3
6	2014+15v/o	PA	9.2×10^{-13}	4.4	4.3	20.0, 22.6
7	2014+25v/o	AR	4.1×10^{-15}	7.3	4.7	14.3, 15.4
8	2014+25v/o	PA	8.1×10^{-13}	5.3	4.3	11.7, 13.9
9	2024+15v/o	PA	8.3×10^{-13}	4.7	4.4	14.7, 17.7
10	2024-T351	PA	1.8×10^{-10}	4.9	2.2	16.4
11	7475+15v/o	AR	3.0×10^{-12}	4.6	3.1	13.4, 13.7
12	7475+15v/o	PA	8.3×10^{-12}	5.1	3.0	13.0, 16.0

AR = All alloys were received as extruded rectangular bars, not further heat treated.

PA = Heat treated according to manufacturers recommendations to obtain conditions equivalent to peak strength (-T6) in the matrix.

* Estimated Value

duplicate values were obtained for most materials. A box is drawn around the largest and smallest values of K_{Ic} obtained because the future direction of the experimental program will be to determine the origins of these fracture toughness values.

Very few measurements have been found in the literature to compare with the results in the Table. Dural Aluminum Composite Company (1) has reported values of K_{Ic} for 2014+10 and 20 v/o SiC PA ranging from 17.7 to 18.8 MPa$\sqrt{m}$, which compares well with our measurements. Data from the same source, dated 1985, gave a value for fracture toughness of 7075+15 v/o SiC PA as 19.7 MPA$\sqrt{M}$, which is nearly 50% more than was obtained with our material.

The material with the lowest value of K_{Ic}, IN9052+15v/o SiC, has already been carefully investigated. The method use to establish K_{Ic} for this composite was similar to that described above, but not exactly the same, in that single edge notched specimens were used in a manually controlled fatigue machine which was periodically stopped to make measurements of the crack length by optical microscopy. It was found during the previous investigation (2,3) that the major contribution to fracture toughness was the energy absorbed in the plastic zone during crack growth. The formation and growth of microvoids along the fracture path was found to absorb a much smaller amount of energy. Some fracture of the largest SiC particles was also found, but that process absorbed even less energy than of void initiation and growth. Virtually no SiC fracture or evidence of microvoid growth was found below the fracture surface; however, the fracture surfaces were found to be rough on a microscopic scale. The interface between SiC and matrix was found to be very strong, so virtually no interfacial separation was observed; thus, voids were not initiated by SiC particles.

Similar analysis of the toughest of the composites, 2014 + 15 v/o SiC Heat Treated to peak strength (Peak Aged), is currently being performed, but is not yet complete. However, the fractography of this material is similar to that of the least tough material, so our expectations are that the crack tip strains will be greater for this tougher material, that the strain distribution within the plastic zone will be similar, and that the plastic zone size will be greater. All of these factors would lead to a larger absorption of energy within the plastic zone of the crack tip for this tougher material. Observations to date indicate some interface cracking between matrix and SiC particles as well as breakage of SiC particles and some matrix cracking. There may be an additional source of toughness in this material, the microcracking, which is another reason a detailed analysis is being conducted.

A correlation has been found between the slope of the crack growth rate curve, s, in eq. (1), and the magnitude of the fracture toughness, Fig. 3. The basis for this correlation has not been found yet, but the existence of the minimum is real.

Fractography - Extensive fractographic examination of both the fatigue and fast fracture zones has been completed, using mainly the scanning electron microscope. Since heat treatment has been found to have an effect on fracture toughness, Table 2, a comparison of the effects of heat treatment on fractography is shown in Figs. 4 and 5.

Fatigue: The general appearance of the fatigue crack region was different between the two heat treatments, left column of Fig. 4. These differences were attributed to the effects of heat treatment even though the stress intensity values are somewnat different; a general evaluation of the

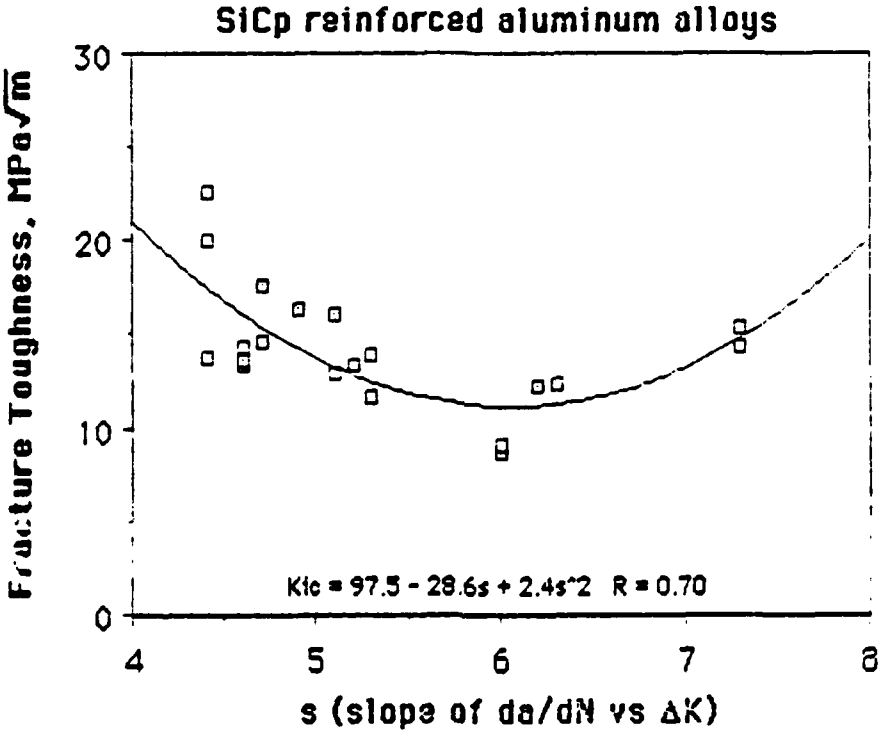

Figure 3. Comparison Between Fracture Toughness Values and Slopes of Fatigue Crack Growth Curves

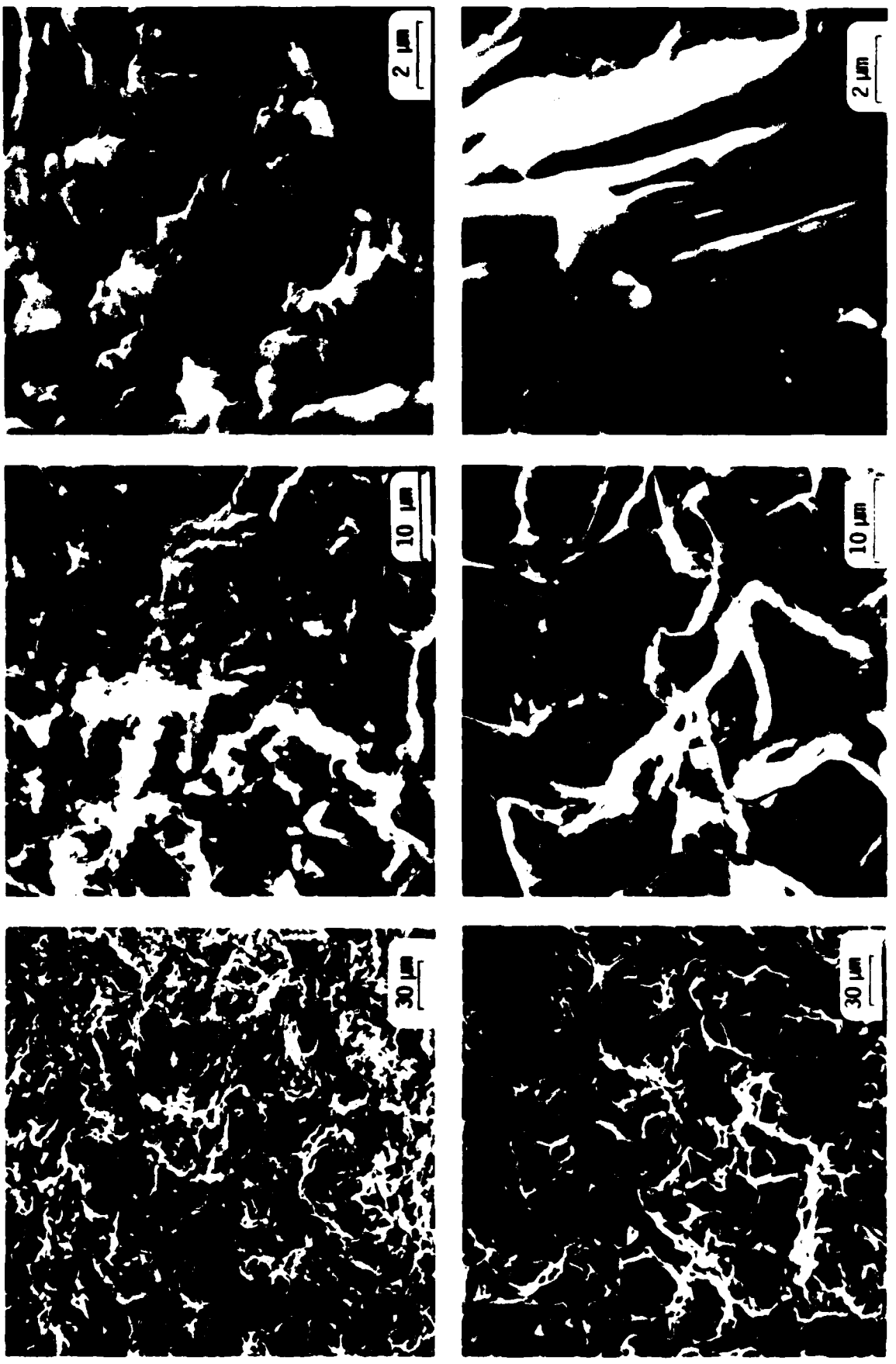

Figure 4. Effect of heat treatment on fatigue fracture appearance. 2014 + 15 v/o SiC: Top row, as-received at ΔK = 6 MPa$\sqrt{m}$; bottom row, peak aged at ΔK = 9 MPa$\sqrt{m}$. Direction of crack growth from left to right.

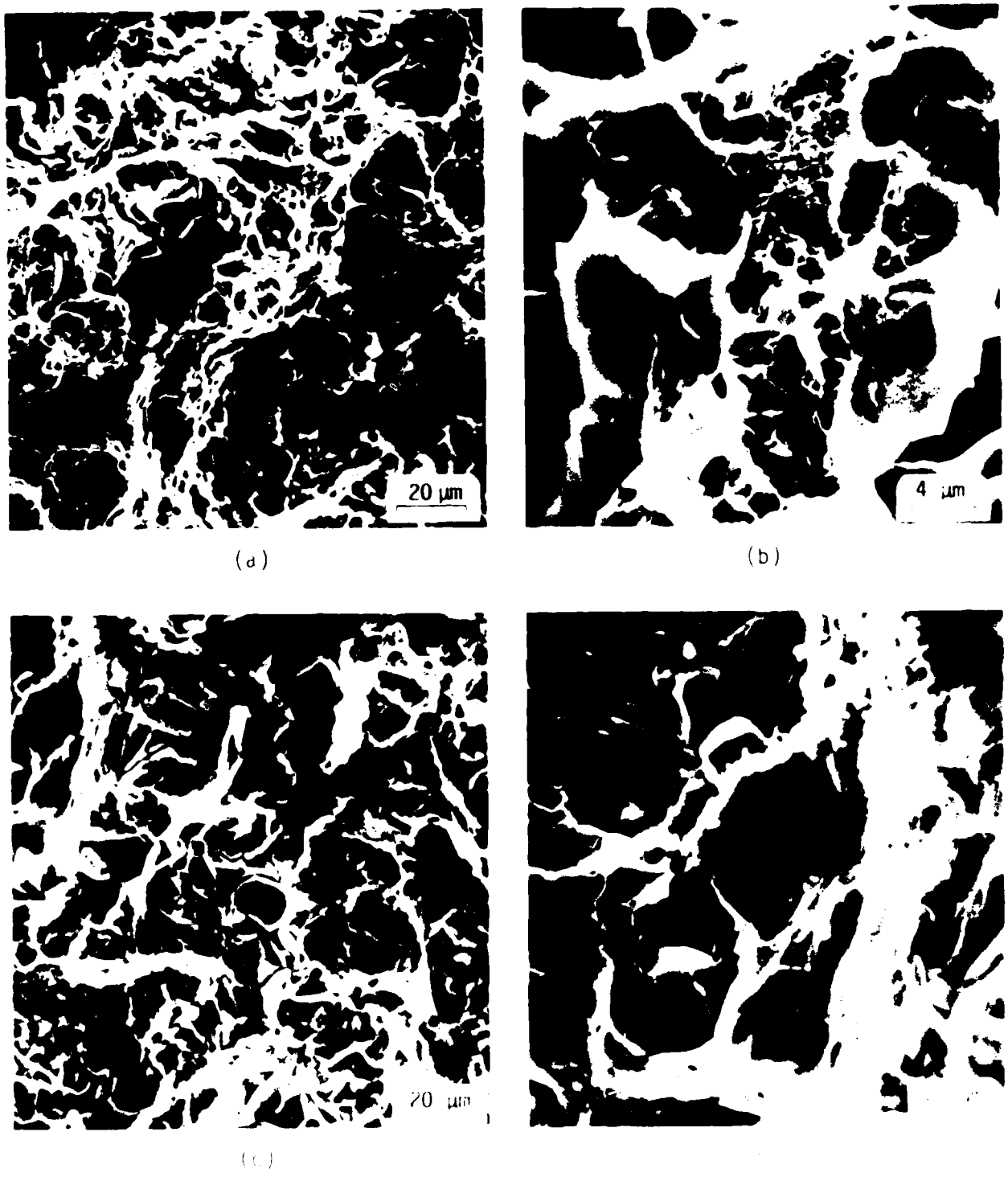

Figure 5.

fracture surfaces indicated this finding to be relatively independent of the stress intensity at which the crack was grown. More evidence of SiC particles was found on the fracture surface of the peak aged composite than for the as extruded material, center column of Fig. 4, and more evidence of "periodic markings" was found on the surface of the peak aged material, right column of the figure.

In general, for both heat treatment conditions, fatigue fracture surfaces revealed the presence of SiC particles. Upon close examination, "periodic markings" could be found on most of the fracture surfaces at most of the stress intensity factors of the tests.

Fast Fracture: Examination of fast fracture surfaces revealed the presence of SiC particles on the surface of all of the composites tested. Patches of very small dimples could usually be found in at least some regions, but these areas were small in most instances and often widely separated, on a microscopic scale.

Ageing treatment was found to have less of an effect on the appearance of the fracture surfaces of the fast fracture region than on the fatigue fracture region. The fast fracture surface of the as extruded material seems to be be rougher than that for peak aged material, Fig. 5, although that is a nonquantitative evaluation.

Cross sections of the fast fracture region are shown in Fig. 6. The low magnification photographs indicate that the as extruded material has greater topography than the peak aged, but the higher magnification photographs appear to show the opposite effect on a microscale. These apparent contradictions in surface roughness have caused us to initiate an effort to measure the

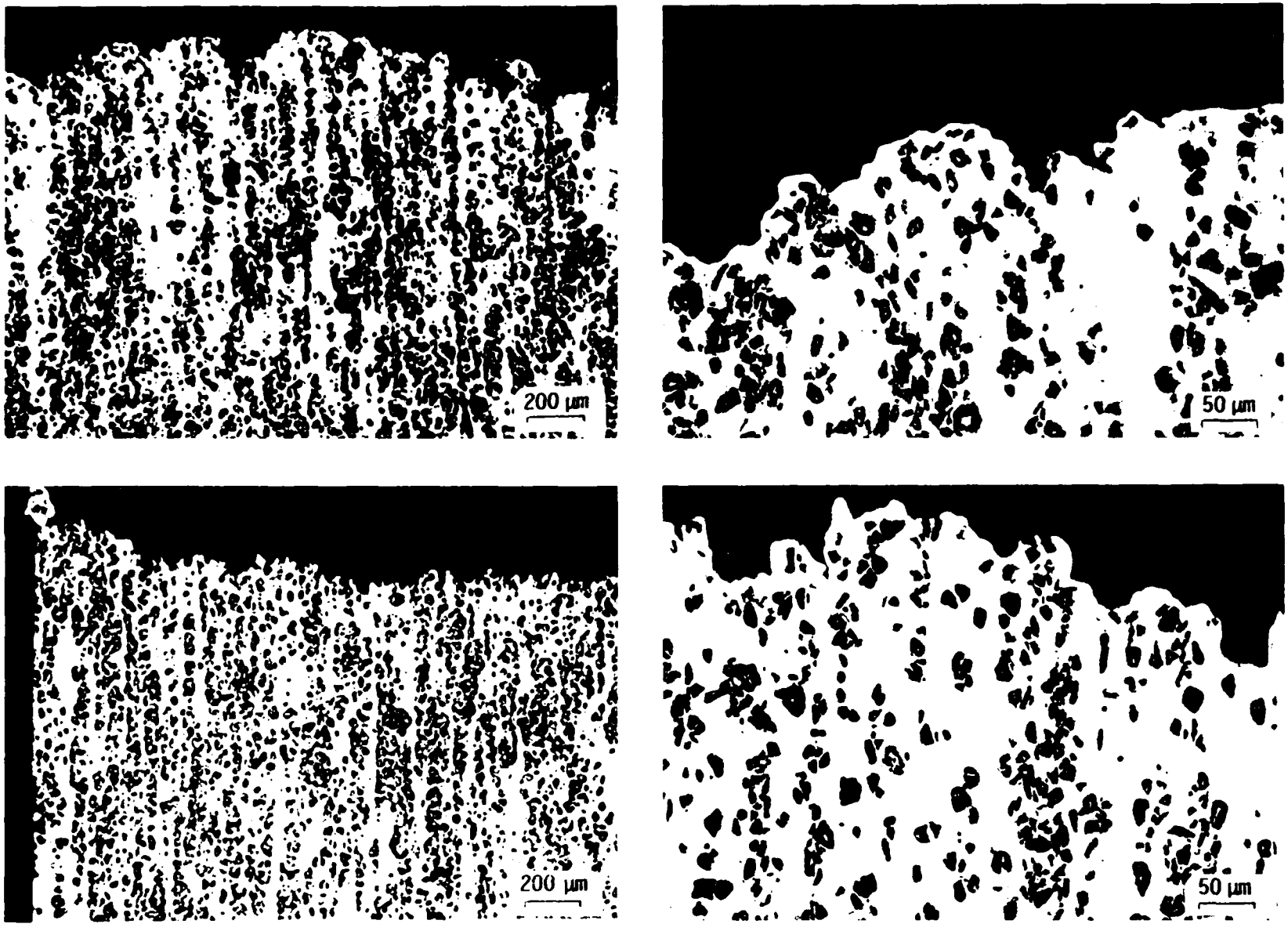

Figure 6. Effect of heat treatment on fast fracture surface roughness. 2014 + 15 v/o SiC cross sections: Top row, as-received, crack growth direction from left to right. Bottom row, peak aged material, crack growth direction into the plane of the photographs.

roughness of these fracture surfaces quantitatively. The measurement technique will be that outlined by E. Underwood (4) and will be accomplished using image processing equipment.

Measurements of the surface roughness will also allow values of the work expended per unit area of crack surface, expressed per unit area normal to the loading axis, to be corrected for actual surface area. The work expended may be computed from values of the crack tip strain and the plastic zone strain distribution using methods derived previously (3). Measurements of these surface roughness quantities are now in progress for several of the composites.

COMPARISONS OF FRACTURE RESULTS:

Comparison of the fracture toughness values of the various composites reveals the effects of some of the variables. Table 3 lists some of the comparisons which may be made. The largest effect appears to be that of heat treatment on 2014 matrix alloy 15 v/o SiC composite, comparison No. 1. The effect of volume fraction SiC on this peak aged matrix is also large, comparison No. 6. For the work hardening matrix Al-4 Mg, the effect of mechanical alloying vs ingot metallurgical fabrication, comparison No. 4, is also significant. Another comparison between mechanical alloying and ingot metallurgy is available by comparing alloys 3 vs 5 and 4 vs 6. In the former comparison, there is little effect of fabrication process, while the latter comparison reveals a large effect.

The large effect on fracture toughness of the peak aging heat treatment for 2014 + 15v/o SiC is not evident for the 25 v/o SiC composite, a curious result; in fact, peak aging the higher SiC material has a reverse (detrimental) effect compared to the lower SiC content material.

TABLE 3

COMPARISONS OF FRACTURE CHARACTERISTICS

Comparison No.	Effect	Alloy No.	vs	Alloy No.	Result (Change in K_{Ic}) MPa$\sqrt{m}$
1	Heat treatment	5		6	14 to 21
2	Heat treatment	11		12	none
3	Heat treatment	7		8	none
4	Mech. alloy+PM vs IM	1		2	9 to 12
5	Vol. Fract SiC, AR	6		7	none
6	Vol Fract SiC, PA	6		8	21 to 13
7	Matrix Alloy, AR	2,3		5,9,11	none
8	Matrix Alloy, PA	4,6,12		6,12	2014+15v/o is best

It is of interest that heat treatment had little effect on the fracture toughness of the 7475 matrix composite, and that composition of the matrix alloy had little effect in general, irrespective of ageing treatment; the exception being 2014. Increasing the SiC content did not alter the fracture toughness significantly.

It is important for the fatigue crack growth rate curve to have as low a slope as possible because the lower the slope, the more damage tolerant is the material. Matrix alloy composition and SiC content did have a marked effect on fatigue properties, particularly the growth rate slope; compare the slopes of the curves for alloys 1 and 2 (s=6) and that of alloys 7 (s=7.3) and 8 (s=5.3) to the remainder of the alloys (s=4.4-5.2). The composite with the work hardening matrix has a somewhat higher slope than is exhibited by other matrix alloys. Conversely, the 2014+15v/o SiC PA composite has a value of s which is about the average of the other materials, yet it exhibits the highest value of fracture toughness, i.e., the increase in fracture toughness has not been at the expense of the fatigue properties. The 2014+25v/o SiC alloy in the as extruded condition has the highest slope (7.3), but average fracture toughness; thus, this composite must be considered one of the least damage tolerant materials. Heat treating this composite to peak aged condition only slightly lowers the fracture toughness, but the slope of the fatigue crack growth rate curve is significantly lowered; the damage tolerance is thus increased.

Values of fatigue crack growth threshold stress intensity factor for the composites (ΔK_{th} = 3 - 4.7MPa$\sqrt{m}$) are significantly greater than those for unreinforced aluminum alloys (ΔK_{th} = 1 - 3), which is a beneficial trait. The material having the largest slope for the fatigue crack growth rate, 2014+25v/o SiC also has the highest threshold, which means there is little

difference between the stress required to grow the fatigue crack and that required to break the specimen.

THE ORIGINS OF FRACTURE TOUGHNESS

Even though our investigation has not yet identified the reasons for the superior fracture toughness of 2014+15 v/o SiC PA, as compared to the rest of the materials, some of the factors which must be considered have been identified. Careful observation of crack growth at near fracture toughness K levels within the scanning electron microscope has been the principal tool for determining the factors which may affect the absorption of energy as the crack grows, supplemented by fractography and metallography of cross sections through the fracture surface. Illustrated in Fig. 7, and listed in the caption, are the events which have been observed to occur during rapid crack growth in these composites.

Of the energy absorption mechanisms listed on Fig. 7, deformation of the material within the plastic zone has been found to dissipate the most energy during fracture. One of the reasons that ceramic reinforced aluminum alloys would be expected to exhibit lower fracture toughness values than the matrix alloy is because of the replacement of part of the plastic zone by non-deformating (ceramic) particles. Thus, the volume of material in the plastic zone adjacent to the crack available to absorb energy is smaller in the composites, which lowers the fracture toughness. Voids formed during the final fracture process are small and only a fraction of the surface is covered by sheets of voids; thus, the energy absorption by this process is small compared to that absorbed in the plastic zone. In addition to directly altering the volume of deforming matrix material within the plastic zone, particles are also expected to change the slip characteristics of this material, thus the

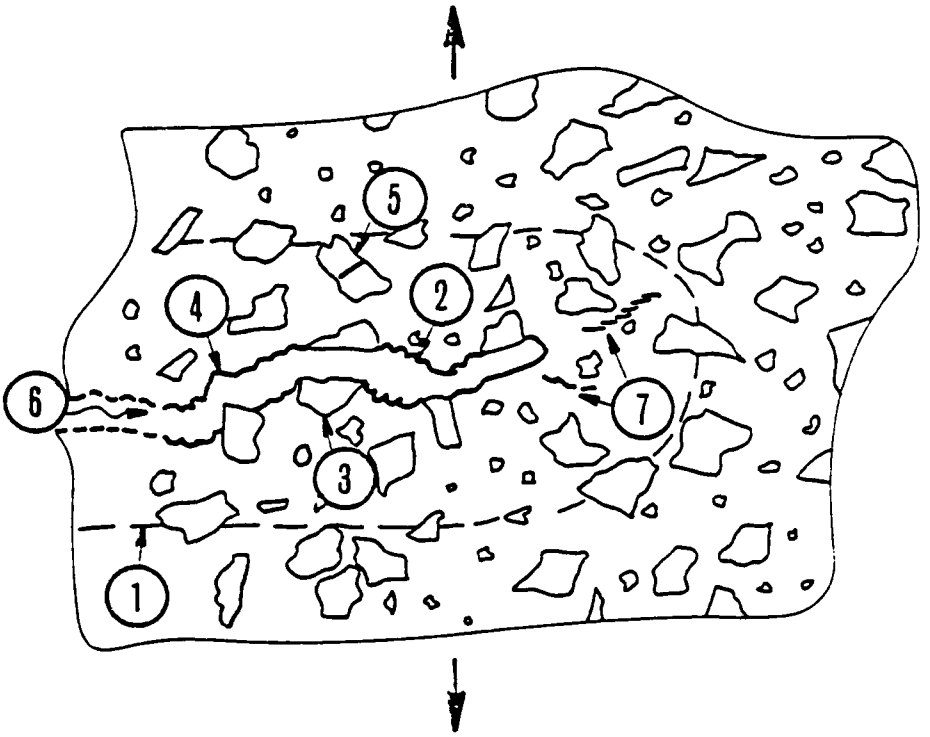

Figure 7. Energy Absorption Mechanisms

1. Deformation of Matrix Within Plastic Zone
2. Formation of Voids Along Fracture Surface
3. Fracture of SiC Particles Along Crack Path
4. Interfacial Separation Between Matrix and SiC
5. Fracture of SiC Within Plastic Zone
6. Torturous Fracture Path Increases Fracture Surface Area. Voids Have Not Been Found to Form Within Plastic Zone Away From Fracture Path
7. Matrix Cracks Near, But Not Contiguous With, The Main Crack

SUMMARY AND CONCLUSIONS

Fatigue crack growth and fracture toughness characteristics of seven particulate silicon carbide reinforced aluminum alloy composites have been determined. Detailed analysis of several of these materials is being conducted to determine the origins of the fatigue and fracture toughness characteristics. The factors contributing to these fracture modes have been established and are being quantitatively determined. Peak aged 2014+15 v/o SiC composite was shown to have the best damage tolerance of all the materials tested. The question of why this combination of alloy and SiC is better than other combinations remains to be answered.

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